47th Tobacco Chemists' Research Conference

October 18-21, 1993

River Terrace Resort & Convention Center
and
The Edgewater Hotel
Gatlinburg, Tennessee USA

Program Booklet — Volume 47

2028896846

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

PROGRAM BOOKLET AND ABSTRACTS - VOLUME 47

47th Tobacco Chemists' Research Conference

October 18-21, 1993

River Terrace Resort & Convention Center

And

The Edgewater Hotel

Gatlinburg, Tennessee USA

HOST

University of Tennessee

2028896847

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

GENERAL PROGRAM

Monday, October 18, 1993

2:00 p.m. - 8:00 p.m.	Registration - River Terrace Ballroom - 2nd Floor Atrium Tower Building
6:30 p.m. - 9:30 p.m.	Reception - River Terrace Ballroom - 2nd Floor Atrium Tower Building Sponsor: U. S. Tobacco Company

Tuesday, October 19, 1993

7:00 a.m.	Program Committee and Session Chair Breakfast - River Terrace Blue Ridge Room - Atrium Tower Building
8:00 a.m. - 4:30 p.m.	Registration - Edgewater Hotel Lobby
8:30 a.m.	Welcome and Opening Remarks - Edgewater Hotel Ballroom (Lobby Level) D. M. Gossett, University of Tennessee
8:45 a.m.	Symposium: Menthol and the Design of Mentholated Cigarettes Introduction: Thomas A Perfetti, Symposium Chairman
10:20 a.m.	Intermission
10:40 a.m.	Session Resumes
12:10 p.m.	Closing Remarks
12:15 p.m.	Lunch
2:00 p.m.	Technical Session (Joint) - Edgewater Hotel Ballroom (Lobby Level) Michael W. Ogden, Presiding
3:20 p.m.	Intermission
3:40 p.m.	Session Resumes
5:00 p.m.	Adjournment
5:15 p.m.	Tobacco Science Editorial Board Meeting - River Terrace Hotel Conference Room - 3rd Floor Atrium Tower Building

2028896848

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

Wednesday, October 20, 1993

7:00 a.m.	TCRC Policy Committee Breakfast - River Terrace Hotel Blue Ridge Room - Atrium Tower Building
8:00 a.m. - 3:30 p.m.	Registration - Edgewater Hotel Lobby
8:40 a.m.	Technical Sessions A and B (Concurrent) - Edgewater Hotel Ballroom Session A - Jimmy H. Bell, Presiding Session B - Larry W. Renfro, Presiding
10:00 a.m.	Intermission
10:30 a.m.	Sessions Resume
11:50 a.m.	Lunch
1:40 p.m.	Technical Session (Joint) - Edgewater Hotel Ballroom R. Hugo Martin, Presiding
3:00 p.m.	Intermission
3:20 p.m.	Session Resumes
4:40 p.m.	Adjournment
4:50 p.m.	Business Meeting - Edgewater Hotel Ballroom
5:45 p.m.	Tobacco Science Council Meeting - River Terrace Conference Room - 3rd Floor Atrium Building
6:30 p.m.	Social Hour - River Terrace Hotel Exhibition Center - 1st Floor Antrium Building
7:30 p.m.	Banquet - River Terrace Hotel Ballroom - 2nd Floor Atrium Building Presentation of the Philip Morris Award for Distinguished Achievement in Tobacco Science Entertainment: Jim Gossett

Thursday, October 21, 1993

7:30 a.m.	ISO TAG Committee Meeting - River Terrace Hotel Conference Room - 3rd Floor Atrium Building
9:00 a.m.	Philip Morris Awardee: Research Highlights - Edgewater Hotel Ballroom
9:50 a.m.	Technical Session (Joint) - Edgewater Hotel Ballroom Byron F. Price, Presiding
10:30 a.m.	Intermission
10:50 a.m.	Session Resumes
11:50 a.m.	Adjournment

Note To Speakers: Room 425 at the Edgewater Hotel is available for slide preview.

2028896849

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

GUESTS' PROGRAM

Monday, October 18, 1993

2:00 p.m. - 8:00 p.m.	Registration
6:30 p.m. - 8:30 p.m.	Reception Sponsor: U. S. Tobacco Company

Tuesday, October 19, 1993

8:00 a.m. - 9:00 a.m.	Continental Breakfast
9:00 a.m.	Bus Departs For Fall Foliage Tour
12:30 p.m.	Return From Fall Foilage Tour (Lunch On Your Own)
	Free Time - Use Trolley Tokens To Shop In Gatlinburg Or Pigeon Forge

Wednesday, October 20, 1993

9:00 a.m.	Breakfast
11:00 a.m. - 3:00 p.m.	Optional Craftsmans Village Tour and Shopping Excursion. Vans will depart at 11:00 a.m. for the Arts and Crafts Community and will be available for return trips to the hotels each hour until 3:00 p.m.; or trolley tokens can be used to visit the Arts and Crafts Community at your leisure.
6:30 p.m.	Social Hour
7:30 p.m.	Banquet - River Terrace Hotel Ballroom - 2nd Floor Atrium Building
	Presentation of the Philip Morris Award for Distinguished Achievement in Tobacco Science
	Entertainment: Jim Gossett

2028896850

47th TOBACCO CHEMISTS' RESEARCH CONFERENCE

TUESDAY MORNING OCTOBER 19, 1993

Joint Session

8:30 a.m.		WELCOME AND OPENING REMARKS D. M. Gossett, Vice President For Agricultural, University Of Tennessee
8:45 a.m.		RECENT ADVANCES IN TOBACCO SCIENCE SYMPOSIUM: **"MENTHOL AND THE DESIGN OF MENTHOLATED CIGARETTES"** INTRODUCTION: Thomas A. Perfetti, Symposium Chairman
8:50 a.m.	#1	MENTHOL: ITS ORIGINS, CHEMISTRY, PHYSIOLOGY AND TOXICOLOGICAL PROPERTIES. Rudolf Hopp, Haarmann & Reimer GmbH, D-37603 Holzminden, Germany
9:20 a.m.	#2	REVIEW OF TECHNOLOGIES RELATING TO MENTHOL USE IN CIGARETTES. August J. Borschke, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA
9:50 a.m.	#3	A HISTORY OF MENTHOLATED CIGARETTES. "THIS SPUD'S FOR YOU." Jack R. Reid, Lorillard Tobacco Company, Greensboro, NC 27420 USA
10:20 a.m.		INTERMISSION
10:40 a.m.	#4	THE CHEMICAL DETERMINATION OF MENTHOL IN THE TOBACCO INDUSTRY FROM AN HISTORICAL PERSPECTIVE. Michael F. Borgerding, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA
11:10 a.m.	#5	THEORETICAL ASPECTS OF MENTHOL MIGRATION AND TRANSFER. Steven A. Wilson, Eastman Chemical Company, Kingsport, TN 37662-5150 USA
11:40 a.m.	#6	EFFECTS OF SOME CIGARETTE CONSTRUCTION PARAMETERS ON MENTHOL MIGRATION AND TRANSFER. Fred W. Best, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA
12:10 p.m.		CLOSING REMARKS
12:15 p.m.		LUNCH

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

TUESDAY AFTERNOON OCTOBER 19, 1993

Joint Session Session Chair: Michael W. Ogden

2:00 p.m. #7 MULTIPLE MEASURES OF PERSONAL ETS EXPOSURE IN A POPULATION-BASED SURVEY OF NONSMOKING WOMEN IN COLUMBUS, OHIO. Michael W. Ogden, Riley A. Davis, K. C. Maiolo, Mitchell F. Stiles, D. L. Heavner, R. B. Hege and W. T. Morgan, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

2:20 p.m. #8 ASSESSMENT OF THE EVERYDAY EXPOSURE TO ENVIRONMENTAL TOBACCO SMOKE (ETS) BY DIFFERENT METHODS. Gerhard Scherer, T. Ruppert, I. Kossien, K. Riedel and F. Adlkofer, Analytisch-Biologisches Forschungslabor, 8000 München 2, Germany

2:40 p.m. #9 CIGARETTE EMISSIONS OF FOURTEEN ETS CONSTITUENTS BY FIVE TYPES OF CIGARETTES ESTIMATED FROM GROWTH CURVES IN A CONTROLLED ENVIRONMENT ROOM. W. S. Rickert, Labstat Incorporated, Kitchener, Ontario N2C 1L3, Canada; M. J. Kaiserman, Health and Welfare, Canada

3:00 p.m. #10 ESTIMATES OF REMOVAL RATES FOR FOURTEEN CONSTITUENTS OF ETS BASED ON A NONLINEAR REGRESSION OF GROWTH CURVES. W. S. Rickert, Labstat Incorporated, Kitchener, Ontario N2C 1L3, Canada; M. J. Kaiserman, Health and Welfare, Canada

3:20 p.m. INTERMISSION

3:40 p.m. #11 SUB-CHRONIC INHALATION STUDY IN RATS, USING AGED AND DILUTED SIDESTREAM SMOKE FROM A REFERENCE CIGARETTE. Chris R. E. Coggins, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

4:00 p.m. #12 *IN VITRO* NITROSATION OF NICOTINE AND SOME OF ITS METABOLITES UNDER PHYSIOLOGICAL CONDITIONS. Mirjana V. Djordjevic, Jingrun Fan, Klaus D. Brunnemann and Dietrich Hoffmann, American Health Foundation, Valhalla, NY 10595 USA

4:20 p.m. #13 NICTOINE UPTAKE FROM SWEDISH MOIST SNUFF. Margareta Curvall and Djuli Ruzeskovic, Reserca AB, S-118 84 Stockholm, Sweden; Gunilla Andersson and Tony Axéll, Lund University, S-214 21 Malmö, Sweden

2028896851

Tobacco Chemists' Research Conference - Volume 47

4:40 p.m. #14 DETERMINATION OF NICOTINE AND COTININE: COMPARISON OF GC AND RADIOIMMUNOASSAY METHODS. Riley A. Davis and Mitchell F. Stiles, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

5:00 p.m. ADJOURNMENT

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

WEDNESDAY MORNING OCTOBER 20, 1993

Session A Session Chair: Jimmy H. Bell

8:40 a.m. #15 AN ALTERNATIVE METHOD FOR THE DETERMINATION OF MENTHOL IN CIGARETTES AND TOBACCO COMPONENTS. Richard D. Hicks and Sharon P. Dunlap, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

9:00 a.m. #16 A PARALLEL STUDY BETWEEN LEAF CHEMISTRY AND PARTICULATE-PHASE SMOKE CHEMISTY. J-Z. Dong, S. C. Moldoveanu and J. H. Lauterbach, Brown & Williamson Tobacco Corporation, Louisville, KY 40210 USA

9:20 a.m. #17 INVESTIGATIONS OF SUCROSE ESTERS BY TANDEM MASS SPECTROMETRY. Terry Sumpter and Nancy J. Jensen, Philip Morris USA, Richmond, VA 23261-6583 USA

9:40 a.m. #18 INCORPORATION OF ^{3}H-NICOTINIC ACID INTO ALKALOIDS OF BURLEY 21, LA BURLEY 21 AND *N. GLAUCA*. Franklin F. Fannin, Nicole Nicholas and Lowell P. Bush, University of Kentucky, Lexington, KY 40546-0091 USA

10:00 a.m. INTERMISSION

10:30 a.m. #19 QUANTITATIVE CHANGES OF CIGARETTE SMOKE COMPONENTS DURING A PUFF. Hideki Takahashi, Yukio Sone, Masao Matsukura and Takeshi Sakaki, Japan Tobacco Incorporated, Midori-ku, Yokohama 227, Japan

10:50 a.m. #20 DETERMINATION OF CADMIUM IN CIGARETTE SMOKE AND ITS POSSIBLE ROLE AS BIOMARKER. Klaus D. Brunnemann, Jonathan E. Cox and Dietrich Hoffmann, American Health Foundation, Valhalla, NY 10595 USA

11:10 a.m. #21 DETERMINATION OF ALDICARB, ALDICARB SULFOXIDE AND ALDICARB SULFONE IN TOBACCO USING HPLC WITH DUAL POST-COLUMN REACTION AND FLUORESCENCE DETECTION. S. S. Yang and I. Smetena, Philip Morris USA, Richmond, VA 23261-6583 USA

11:30 a.m. #22 NICOTINE AND CARBOXYLIC ACIDS: STRUCTURE AND SOLUTION DYNAMICS, A MULTINUCLEAR NMR STUDY. William S. Caldwell, William M. Coleman and Patricia Martin, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

2589688202

11:50 a.m. LUNCH

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

WEDNESDAY MORNING OCTOBER 20, 1993

Session B Session Chair: Larry W. Renfro

8:40 a.m. #23 A MATHEMATICAL MODEL OF PYROLYSIS PROCESSES IN A SMOLDERING CIGARETTE. Peishi Chen and Sung-Chul Yi, Philip Morris USA, Richmond, VA 23261-6583 USA

9:00 a.m. #24 NUMERICAL RESOLUTION OF 2-D COMBUSTION FRONT PROPAGATION BY ORTHOGONAL ADAPTIVE MESH TECHNIQUE. Sung-Chul Yi, Philip Morris USA, Richmond, VA 23261-6583 USA

9:20 a.m. #25 A COMPARATIVE IGNITION PROPENSITY STUDY VIA INFRARED IMAGING ANALYSIS. Marian Joyce, Tim Nestor and Stephen Sears, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

9:40 a.m. #26 ORGANIC POTASSIUM. ITS ROLE IN CONTROLLING THE RATE OF BURN AND 'TAR' CONTENT OF TOBACCO PRODUCTS. James W. Horler, Professional Services, Wells, Somerset BA5 1NY, England; Nicholas Baskévitch, LTR Industries, Le Mans, France

10:00 a.m. INTERMISSION

10:30 a.m. #27 THE EFFECT OF CELLULOSE ACETATE FIBER AND FILTER PARAMETERS ON ROD FIRMNESS. Cheryl E. Johnson, Hoechst Celanese Corporation, Charlotte, NC 28232 USA

10:50 a.m. #28 THE ROLE OF PAPER FILTERS IN THE CHANGING CIGARETTE MARKET. Richard J. K. Shepherd, FIL International Limited, Harpenden, Hertfordshire AL5 4SJ, England

11:10 a.m. #29 PREDICTION OF FILTER CIGARETTE DESIGN UNDER RESTRICTED CONDITIONS. Young-Hoh Kim, Kwang-Soo Rhim and Yong-Tae Kim, Korea Ginseng and Tobacco Research Institute, Yoosungku, Daejeonshi, Korea 305-345

11:30 a.m. #30 THE EFFECT OF VENTILATION ON BURN RATES. Lydia J. Holt and Larry W. Renfro, Eastman Chemical Company, Kingsport, TN 37622-5150 USA

11:50 a.m. LUNCH

2028896853

WEDNESDAY AFTERNOON OCTOBER 20, 1993

Joint Session Session Chair: R. Hugo Martin

1:40 p.m. #31 SOLUTION CHEMISTRY OF PSEUDOOXYNICOTINE AND 2'-CYANONICOTINE. A HIGH FIELD NMR STUDY. Grayland P. Dobson and William S. Caldwell, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

2:00 p.m. #32 DETERMINATION OF NICOTINE-1',2'- AND 1',5'-IMINIUM IONS IN AQUEOUS SOLUTIONS. Gary D. Byrd, G. M. Dull, Grayland P. Dobson and William S. Caldwell, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

2:20 p.m. #33 IDENTIFICATION AND QUANTIFICATION OF TOBACCO-SPECIFIC NITROSAMINE PRECURSORS IN AIR-CURED TOBACCO. Harold R. Burton and Xiaochen Wei, University of Kentucky, Lexington, KY 40546 USA; William S. Caldwell, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

2:40 p.m. #34 DIFFERENCES IN COMPOSITION OF AIR-CURED BURLEY TOBACCO USING MODIFIED FIELD CURING STRUCTURES. 1. CHANGES IN NITROGENOUS CONSTITUENTS. Harold R. Burton, N. K. Dye and L. Walton, University of Kentucky, Lexington, KY 40546-0091 USA

3:00 p.m. INTERMISSION

3:20 p.m. #35 NOVEL DITERPENE GLYCOSIDES FROM *NICOTIANA* SPECIES. Tetsuya Tobita and Y. Shinozaki, Japan Tobacco Incorporated, Tokyo 130, Japan; H. Takahashi, M. Mizutani and T. Matsuzaki, Japan Tobacco Incorporated, Yokohama, Kanagawa 227, Japan

3:40 p.m. #36 VOLATILE CONSTITUENTS FROM FLOWERS OF SELECTED *NICOTIANA* SPECIES. William S. Schlotzhauer, Robert J. Horvat and Orestes T. Chortyk, United States Department of Agricultural, Athens, GA 30613 USA; D. Michael Jackson, United States Department of Agricultural, Oxford, NC 27565-1168 USA

4:00 p.m. #37 EVALUATION OF LOW NICOTINE FLUE-CURED TOBACCO IN BLENDED CIGARETTES. Willard W. Weeks, North Carolina State University, Raleigh, NC 27695 USA; Verne A. Sisson, United States Department of Agricultural, Oxford, NC 27565 USA

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

4:20 p.m. #38 NATURAL PRODUCTS AS SOURCES OF AROMA AND FLAVOR INGREDIENTS. William M. Coleman, J. L. White, T. A. Perfetti and W. T. Morgan, R. J. Reynolds Tobacco Company, Winston-Salem, NC 27102 USA

4:40 p.m. ADJOURNMENT

4:50 p.m. BUSINESS MEETING

5:20 p.m. TOBACCO SCIENCE COUNCIL MEETING

2028896854

THURSDAY MORNING OCTOBER 21, 1993

Joint Session Session Chair: Byron F. Price

9:00 a.m. PHILIP MORRIS AWARDEE, RESEARCH HIGHLIGHTS

9:50 a.m. #39 A NEW ANALYTICAL METHOD FOR THE DETERMINATION OF NITRITE IN NANOGRAM LEVELS IN TOBACCO. Alex Christakopoulos, Ann Palmqvist and Inger Wahlberg, Reserca AB, S-118 84 Stockholm, Sweden

10:10 a.m. #40 INFLUENCE OF TOBACCO NITRATE CONTENT ON THE YIELDS OF SELECTED MAINSTREAM SMOKE COMPONENTS. Anthony R. Tricker, Verband der Cigaretten Industrie, 5300 Bonn 3, Germany; Gerhard Scherer and F. Adlkofer, Analytisch-Biologisches Forschungslabor, 8000 München 2, Germany

10:30 a.m. INTERMISSION

10:50 a.m. #41 A METHOD FOR THE DETERMINATION OF POLYPHENOLS IN TOBACCO BY HPLC AND ITS APPLICATION TO THE ANALYSIS OF VARIOUS TOBACCO TYPES. Steve J. Stotesbury, British-American Tobacco Company Limted, Southampton SO9 1PE, England

11:10 a.m. #42 THE POLYPHENOLS OF *NICOTIANA*. Maurice E. Snook, Ray F. Severson, Orestes T. Chortyk, United States Department of Agricultural, Athens, GA 30613 USA; Verne A. Sisson, United States Department of Agricultural, Oxford, NC 27565-1168 USA

11:30 a.m. #43 AN IMPROVED INSTRUMENT AND DATA ANALYSIS APPROACH FOR THE STUDY OF HOT COLLAPSE OF CIGARETTE FILTERS. Lance J. Deutsch, Hoechst Celanese Corporation, Charlotte, NC 28232 USA

11:50 a.m. ADJOURNMENT

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

TUESDAY MORNING

Joint Session

Thomas A. Perfetti, Presiding

1
8:50 a.m.
Tuesday

MENTHOL: ITS ORIGINS, CHEMISTRY, PHYSIOLOGY AND TOXICOLOGICAL PROPERTIES. Rudolf Hopp, Haarmann & Reimer GmbH, Corporate Research, Rumohrtalstrasse 1, D-37603 Holzminden, Germany

Mint flavor (both peppermint and spearmint) represents the third largest flavor type worldwide; whereas, menthol ranks second as an aroma chemical after vanillin. The review starts with some historical and economic facts regarding the occurrence, production and use of peppermint and menthol. It is shown that the major types of peppermint oil are different with respect to flavor profile and chemical composition. In the biosynthesis of the main constituents, geranyl pyrophosphate is first cyclized to (-)-limonene and then transformed to the oxygenated menthanes. The high enantioselectivity of this pathway is confirmed by chiral GC. The stereochemistry of menthol is a good example of structure-odor relationship. Preparation and sensory impressions of the eight enantiomers are described. Though cornmint oil (*mentha arvensis*) is still the main source of (-)-menthol, many synthetic processes have been developed, starting from natural optically active raw materials or from basic organic chemicals. The few processes which achieved commercial importance are outlined. The major sensory property of (-)-menthol is its cooling sensation on the skin or in the mouth. This effect results from a stimulating action on peripheral cold receptors. Due to a demand for nonodorous substances possessing a longer lasting cooling effect, some new cooling agents (mostly menthol analogues) have been developed and marketed more recently. The reviewed toxicological and biological studies on menthol show that it is nontoxic at levels used in commercial products, not carcinogenic and easily biodegradable.

2
9:20 a.m.
Tuesday

REVIEW OF TECHNOLOGIES RELATING TO MENTHOL USE IN CIGARETTES. August J. Borschke, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

The patent art relating to the use of menthol in smoking articles for the period from 1924 through 1992 has been reviewed. The majority of the patent documents reviewed

2028896855

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

has been awarded to cigarette manufacturing companies. However, a significant number of patent documents also are owned by companies which supply materials or machinery for cigarette manufacture. Approximately 100 patent documents which have been reviewed have disclosed methods for applying menthol to tobacco, methods for incorporating menthol into smoking article components, and various menthol compositions. Typically, menthol is applied by spraying solutions or dispersions containing menthol onto tobacco. Menthol has been incorporated into papers used to manufacture cigarettes, into cigarette rods and into cigarette filters. Menthol also has been incorporated into cigarette packaging materials. Menthol compositions often use precursor compounds which release menthol when cigarettes incorporating such compounds are smoked.

3
9:50 a.m.
Tuesday

A HISTORY OF MENTHOLATED CIGARETTES. "THIS SPUD'S FOR YOU." Jack R. Reid, Lorillard Tobacco Company, P. O. Box 21688, Greensboro, North Carolina 27420 USA

In the late 1920's, *Spud* (the first mentholated cigarette) was marketed. Following early entries of *Penguin* and *Kool* brands in the 1930's, the market share maintained a consistent 2% up until the introduction of filters in the mid 1950's. *Salem*, then other, filtered mentholated cigarettes were offered to the public, and the market share of mentholated cigarettes increased, finally reaching a peak of about 30% in the mid 1970's. Currently mentholated cigarettes account for about 25% of the total cigarette sales.

10:20 a.m. INTERMISSION

4
10:40 a.m.
Tuesday

THE CHEMICAL DETERMINATION OF MENTHOL IN THE TOBACCO INDUSTRY FROM AN HISTORICAL PERSPECTIVE. Michael F. Borgerding, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

A general overview of the apparatus, procedures and instrumentation applied to the determination of menthol as it relates to the tobacco industry is presented. Evolution and refinement of chemical analyses for the determination of menthol in tobacco, cigarette smoke, cigarette components and flavoring materials are reviewed. Particular emphasis is placed on summarizing and comparing reported method validation data.

2028896856

5
11:10 a.m.
Tuesday

THEORETICAL ASPECTS OF MENTHOL MIGRATION AND TRANSFER. Steven A. Wilson, Eastman Chemical Company, P. O. Box 1972, Kingsport, Tennessee 37662-5150 USA

The behavior of menthol in cigarettes during storage and smoking has been extensively studied because of the popularity and the unique properties of menthol. Most studies have involved empirical measurements of menthol's migration during storage and transfers during smoking. It is well known that menthol has significant migration during storage because of its volatility. In general, the studies have found that menthol seeks a balance between the two major components of the cigarette, the tobacco column and cellulose acetate filter. This balance is influenced by the cigarette construction parameters and has an impact on the transfers into smoke. Studies have shown that the transfer from the tobacco column during smoking is constant with time, but the transfer from cellulose acetate filters changes with time due to diffusion of the menthol into plasticized fibers of the filter. Some researchers have formulated and tested theories for these behaviors and have been successful at making reasonable predictions. A review of the general behavior and current theories of the processes occurring within mentholated cigarettes would be valuable because it allows one to link the results and conclusions from the various studies. This paper discusses the current theories that describe the characteristics of menthol's migration and transfer in cigarettes.

6
11:40 a.m.
Tuesday

EFFECTS OF SOME CIGARETTE CONSTRUCTION PARAMETERS ON MENTHOL MIGRATION AND TRANSFER. Fred W. Best, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

In the early days of mentholated filter cigarettes, 'tar' yield was high and menthol transfer to mainstream smoke was fairly efficient. Filters were undiluted and generally inefficient. Early fate studies determined the distribution of menthol to mainstream smoke and filter and/or butt to be greater than 50% of the applied menthol. But modifications to cigarettes to decrease 'tar' also reduced menthol transfer efficiency. Only about 10% (or less) of the menthol in contemporary cigarettes transfers to mainstream smoke. In attempts to find ways to achieve 'tar' reductions with minimum impact on menthol transfer efficiency, the effect of various cigarette components and variations in cigarette construction on menthol transfer has been extensively studied. Filters exert a major influence on menthol transfer to smoke. Effects of filters and

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

variations in filter construction on menthol transfer to mainstream smoke have been the parameters on which most studies focused. This report will review some of the previously reported research and discuss some previously unreported findings. Effects of a tobacco humectant, propylene glycol, and of five different individual tobacco blend components on menthol migration and transfer are also discussed.

12:10 p.m. SUMMARY AND CLOSING REMARKS

12:15 p.m. LUNCH

2028896857

TUESDAY AFTERNOON

Joint Session

Michael W. Ogden, Presiding

7
2:00 p.m.
Tuesday

MULTIPLE MEASURES OF PERSONAL ETS EXPOSURE IN A POPULATION-BASED SURVEY OF NONSMOKING WOMEN IN COLUMBUS, OHIO. Michael W. Ogden, Riley A. Davis, K. C. Maiolo, Mitchell F. Stiles, D. L. Heavner, R. B. Hege and W. T. Morgan, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

Environmental tobacco smoke (ETS) exposure was assessed continuously over a one-week period in a group of 96 nonsmoking, married women in Columbus, Ohio. Markers used to estimate ETS exposure were airborne nicotine and 3-ethenylpyridine (3-EP), and salivary cotinine. For purposes of assessing relative ETS exposure, the women were categorized according to husband's smoking status (e.g., ETS-exposed if husband smokes). The relative exposures measured were: 8.6 for cotinine; 13.4 for nicotine; and 22.0 for 3-EP. These relative exposures are significantly larger than those assumed in recent ETS risk assessments and tend to question the validity of risk estimate corrections for so-called "background exposure." The same ratios measured for employed subjects indicate that workplace exposure is tenfold lower than home exposure due to living with a smoker. In addition, these data also suggest that cotinine alone may be an inadequate marker for estimating ETS exposure in the general population. Median concentrations (all women) were found to be: 0.15 $\mu g/m^3$ (3-EP); 0.43 $\mu g/m^3$ (nicotine); and 0.8 ng/ml (salivary cotinine).

8
2:20 p.m.
Tuesday

ASSESSMENT OF THE EVERYDAY EXPOSURE TO ENVIRONMENTAL TOBACCO SMOKE (ETS) BY DIFFERENT METHODS. Gerhard Scherer, T. Ruppert, I. Kossien, K. Riedel and F. Adlkofer, Analytisch-Biologisches Forschungslabor, Goethestr. 20, 8000 München 2, Germany

A household study is in progress, in which the everyday ETS exposure is assessed by questionnaires, air monitoring in homes, personal nicotine samplers and biomonitoring. The participating household members keep diaries on their tobacco smoke exposure (active smoking or ETS exposure) over

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

an eight-day period. During that period they wear nicotine personal diffusion samplers. On the first day and the last day of the study, the participants collect saliva and spot urine samples for the determination of nicotine and cotinine. A three hour air monitoring in the living room is performed in the evening when all household members are present. Air samples were collected for the determination of carbon monoxide, respirable suspended particles (RSP), solanesol, nicotine and 3-ethenylpyridine. An intermediate evaluation of the data from eight smoking homes (average: 1.5 smokers per home) revealed the following results: The average number of cigarettes smoked in the three hour monitoring period was 7.6 (range: 3 to 20). The mean concentrations (ranges) of carbon monoxide, nicotine and RSP amounted to 0.7 (0 to 1.4) ppm, 7.9 (0.9 to 30.4) $\mu g/m^3$, and 247 (69 to 581) $\mu g/m^3$, respectively. The nonsmokers reported an average ETS exposure time of 3.2 (0 to 8.4) hours per day. The amount of nicotine on the personal samplers revealed an average nicotine exposure concentration for the nonsmokers of 1.0 (0.1 to 4.1) $\mu g/m^3$ during the course of the study. Based on the actual self-reported ETS exposure times, the nicotine levels were 4.5 (0.7 to 18.3) $\mu g/m^3$.

9
2:40 p.m.
Tuesday

CIGARETTE EMISSIONS OF FOURTEEN ETS CONSTITUENTS BY FIVE TYPES OF CIGARETTES ESTIMATED FROM GROWTH CURVES IN A CONTROLLED ENVIRONMENT ROOM.

W. S. Rickert, Labstat Incorporated, 262 Manitou Drive, Kitchener, Ontario N2C 1L3, Canada; M. J. Kaiserman, Health and Welfare, Canada

It is often assumed that environmental emissions are largely independent of cigarette type and smoking conditions. This hypothesis has been investigated in a series of controlled environment room (CER) experiments which examined the effect of brand (5 types) lighting (on/off), puff volume (35/50 ml) and ventilation of emissions. In this project, a single port smoking machine was used to generate sidestream smoke at a constant rate in a 37 m^3 CER for a period of five hours. Constituent concentrations were monitored for the course of the experiment and equilibrium concentrations (A_{eq}) estimated by nonlinear regression assuming the model $C_t = A_{eq} (1 - e^{Cat})$. Values for A_{eq} for each constituent were converted to emissions per cigarette based on the estimated effective removal rate (C_at), the room volume and the number of cigarettes smoked. Results for CO averaged from 85 to 113 mg per cigarette which is about twice that determined by conventional means. NO emissions ranged from 50 to 85 μmoles/cigarette and formaldehyde from 880 to 2,410 μg/cigarette which is also considerably in excess of values determined using the BAT fishtail chamber. Brand dependency for other carbonyls (acetaldehyde, propionaldehyde,

2028896858

crotonaldehyde, butyraldehyde, acetone, acrolein and methyl ethyl ketone) was not as pronounced. Emissions of benzo[a]pyrene ranged from 107 to 139 ng/cigarette; hydrogen cyanide from 74 to 149 μg/cigarette and benzene from 523 to 978 ng/cigarette. Cadmium emissions ranged from 87 to 167 ng/cigarette based on the average level observed over the course of the 5 hour experiment. In general, neither lighting nor ventilation were significant determinants of cigarette emissions but with some significant exceptions. The puff volume effect was significant for some variables but not large.

10
3:00 p.m.
Tuesday

ESTIMATES OF REMOVAL RATES FOR FOURTEEN CONSTITUENTS OF ETS BASED ON A NONLINEAR REGRESSION OF GROWTH CURVES.

W. S. Rickert, Labstat Incorporated, 262 Manitou Drive, Kitchener, Ontario N2C 1L3, Canada; M. J. Kaiserman, Health and Welfare, Canada

The usefulness of environmental tobacco smoke (ETS) surrogates, such as solanesol, depends upon defining the relationship between the surrogate and other ETS constituents. For a system at equilibrium this does not pose a problem. However, equilibrium is rarely achieved outside of the controlled environment room (CER) due to uncontrolled or unknown factors such as intermittent and variable emissions and differing removal rates. We have examined the removal rate question in a series of 120 controlled environment room experiments. Factors examined include puff volume (35/50 ml), lighting (on/off), brand type (5) and ventilation (2 levels). The concentration of various ETS constituents was monitored for five hours in a 37 m^3 CER with a constant source of sidestream smoke. Estimates of removal rates for each constituent were obtained using nonlinear regression assuming a model of the form $C_t = A_{eq} (1 - e^{Cat})$. In general, removal rate was a function of ventilation but the relationship was not simple for compounds such as acrolein and formaldehyde. Point estimates for C_a at the lowest ventilation rate in units of air exchanges per hour (ACH) are CO, 0.395; NO, 0.581; formaldehyde, 1.34; acetaldehyde, 0.484; propionaldehyde, 0.607; crotonaldehyde, 0.693; butyraldehyde, 0.758; acetone, 0.480; acrolein, 0.240; methyl ethyl ketone, 0.679; hydrogen cyanide, 0.679; benzene, 0.461; toluene, 0.440; and benzo[a]pyrene, 0.495. Results from the higher ventilation rate will be presented and the implications for estimation by surrogate discussed.

3:20 p.m. INTERMISSION

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

11
3:40 p.m.
Tuesday

SUB-CHRONIC INHALATION STUDY IN RATS, USING AGED AND DILUTED SIDESTREAM SMOKE FROM A REFERENCE CIGARETTE. Chris R. E. Coggins, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

Male Sprague-Dawley rats were exposed six hours per day, five days per week for up to 13 weeks to aged and diluted sidestream smoke (ADSS), used as a surrogate for environmental tobacco smoke (ETS), at concentrations of 0.1 ("typical"), 1 ("extreme") or 10 ("exaggerated") milligrams of particulates per cubic meter. Animals were exposed nose-only, inside whole-body chambers, to ADSS from the 1R4F reference cigarette. The target particulate concentrations were achieved; at the exaggerated exposure they resulted in carbon monoxide (CO) concentrations in excess of 50 ppm. The only histopathological response observed was slight to mild epithelial hyperplasia in the rostral nasal cavity in the exaggerated exposure group only. No effects were noted at extreme or typical exposures. The changes were similar in animals killed after 4, 28 or 90 days, and were also similar to those noted in an earlier experiment with only 14 days duration, indicating that the change does not progress with increased exposure duration from 4 to 90 days. The nasal change was absent in a subgroup of animals kept without further treatment for an additional 90 days, indicating complete reversibility.

12
4:00 p.m.
Tuesday

IN VITRO NITROSATION OF NICOTINE AND SOME OF ITS METABOLITES UNDER PHYSIOLOGICAL CONDITIONS. Miriana V. Djordjevic, Jingrun Fan, Klaus D. Brunnemann and Dietrich Hoffmann, American Health Foundation, 1 Dana Road, Valhalla, New York 10595 USA

The major *Nicotiana tabacum* alkaloid nicotine is a precursor of five tobacco-specific nitrosamines (TSNA) that occur in tobacco. Three of these TSNA are also found in cigarette smoke either being transferred from tobacco or formed during smoking. To address the question whether additional amounts of TSNA are formed endogenously in tobacco users, an *in vitro* nitrosation study of nicotine and its major metabolites (cotinine and cotinine acid) was carried out under different pH conditions at 37°C with or without thiocyanate. The reaction time was 30 minutes and 24 hours. The major nitrosation products of nicotine at pH 1.5 were 4-(methylnitrosamino)-4-(3-pyridyl)butanal (up to 5.8% of nicotine) followed by N'-nitrosonornicotine (NNN; up to 0.77%) and 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone

(up to 0.16%). At pH 5.5 and pH 8, both the yields and the composition of TSNA changed dramatically with NNN being the most abundant TSNA at pH 8 but amounting only to 0.05% of nicotine. Interestingly, the major nitrosation products of cotinine were N'-nitrosoanatabine and N'-nitrosoanabasine; whereas, cotinine acid gave rise to 4-(methylnitrosamino)-4-(3-pyridyl)butyric acid (up to 100% of cotinine in the presence of the catalyst). The levels and the composition of nitrosation products of nicotine, cotinine, and cotinine acid will be discussed in greater detail.

13
4:20 p.m.
Tuesday

NICOTINE UPTAKE FROM SWEDISH MOIST SNUFF. Margareta Curvall and Djuli Ruzeskovic, Reserca AB, S-118 84 Stockholm, Sweden; Gunilla Andersson and Tony Axéll, Lund University, Department of Oral Surgery and Oral Medicine, S-214 21 Malmö, Sweden

This study was performed in order to investigate whether users of Swedish moist snuff (snus) compensate for the reduced delivery of nicotine when switching from their ordinary brand (Brand 1) to a new brand containing half the amount of nicotine (Brand 2). The subjects who took part in the study were 24 healthy male volunteers. They consumed *ad libitum* Brand 1 during the first two weeks and Brand 2 for the next ten weeks. The daily consumption in terms of grams snus per day and hours per day was recorded for seven days during week 1, 2, 4, 8 and 12. In each of these weeks 24 hour urine samples were collected on day 6 and saliva spot samples were gathered on day 7. Total amount of nicotine and its seven main metabolities in 24 hour urine samples and cotinine in saliva samples were quantified. The nicotine dose was estimated as the total amount of nicotine and metabolites excreted during 24 hours expressed as nicotine equivalents. There was no change in number of hours of daily consumption, while there was a slight increase in total amount of snus consumed per day (2.5 grams) after switching from Brand 1 to Brand 2. There was a significant decrease in saliva cotinine level as well as in nicotine dose after switching from Brand 1 to Brand 2, i.e., the average salivary cotinine concentration fell from 336 to 153 ng/ml and the average nicotine dose from 25.2 to 14.4 mg, respectively. These results were compared with the results from a reference group comprising 18 subjects who had consumed Brand 2 for more than 6 months. Detailed data on consumption, nicotine dose, salivary cotinine and nicotine disposition will be presented.

2028689859

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

14
4:40 p.m.
Tuesday

DETERMINATION OF NICOTINE AND COTININE: COMPARISON OF GC AND RADIOIMMUNOASSAY METHODS. Riley A. Davis and Mitchell F. Stiles, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

Measurement of nicotine and/or cotinine in body fluids is used to assess smoking status, maintenance of smoking cessation, metabolic disposition of nicotine/cotinine, and nicotine absorbed from other sources. Instrumental methods chosen for the analysis of nicotine and cotinine depend on the function of the assessment(s). Both instrumentation and lengthy sample preparation lead to increased cost per analysis. To increase the efficiency of sample analysis, we investigated the use of radioimmunoassay (RIA) and compared its performance against GC-NPD. Our results indicated that the sensitivity of the RIA rivaled GC-NPD in all matrices. RIA is not as specific as the GC method for determining nicotine and cotinine in urine since the polyclonal antibodies used in the RIA cross-react with nicotine metabolites including nicotine-derived glucuronides. Our studies demonstrated that RIA offers the following advantages: a) sensitivity, b) little or no sample preparation, c) ease of implementation, and d) cost savings. Results of RIA validation and its application will be discussed also.

5:00 p.m. ADJOURNMENT

02089688

WEDNESDAY MORNING

Session A

Jimmy H. Bell, Presiding

15
8:40 a.m.
Wednesday

AN ALTERNATIVE METHOD FOR THE DETERMINATION OF MENTHOL IN CIGARETTES AND TOBACCO COMPONENTS. Richard D. Hicks and Sharon P. Dunlap, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

A relatively simple, straightforward and rugged method has been developed to determine menthol in cigarettes and tobacco components. Menthol in cigarettes and tobacco components was quantitatively extracted using 3A alcohol (ethanol) in a heated shaker water bath at 60°C for one hour at 200 rpm. The extract was then analyzed by capillary gas chromatography. During the development of the method, various solvents, flask sizes, and solvent volumes were explored to determine optimum extraction conditions. The method was validated for menthol in the filter, rod, whole cigarette and cut filler tobacco. Samples in the 0.1 to 2.0 weight percent range were used for method validation and overall method reproducibility (relative standard deviation) was less than 3%. During the validation process, samples were analyzed simultaneously by this extraction technique and by the standard AOAC (Association of Official Analytical Chemists) steam distillation technique for comparison. This new method has several distinct advantages over the AOAC technique. The extraction method is relatively simple to execute, requires no extensive training of the analyst, and procedural hazards are minimized. Unlike the distillation technique, constant monitoring is not necessary and the analyst is available to perform other laboratory tasks.

16
9:00 a.m.
Wednesday

A PARALLEL STUDY BETWEEN LEAF CHEMISTRY AND PARTICULATE-PHASE SMOKE CHEMISTRY. J-Z. Dong, S. C. Moldoveanu and J. H. Lauterbach, Brown & Williamson Tobacco Corporation, I. W. Hughes Technical Center, 1600 West Hill Street, Louisville, Kentucky 40210 USA

GC/MS scan techniques provide a convenient way for "fingerprinting" complex mixtures. We have used two main scan techniques in our laboratory for the characterization of tobacco leaf. One technique consists of extraction of the tobacco with a very efficient solvent, followed by GC/MS

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

analysis. This technique allows the analysis of the more volatile compounds, starting with low molecular weight ketones and extending up to neophytadiene or scopoletin. The second technique consists of the simultaneous extraction and silylation of the semivolatile compounds in tobacco. This technique allows the analysis of compounds extending from lactic acid up to sucrose and chlorogenic acid. We have reported on both these techniques at the 46th Tobacco Chemists' Research Conference. We have now applied both techniques for the analysis of the particulate phase of cigarette smoke. Single-grade flue-cured, burley, and oriental cigarettes were smoked with a Borgwaldt RM-20 smoking machine. The smoke from 20 cigarettes was collected on a 92 mm Cambridge pad. Sections of the pad were then extracted and analyzed by the same techniques that were used to analyze tobacco from these cigarettes. Correlations between the tobacco and smoke chromatograms were surprisingly good.

17
9:20 a.m.
Wednesday

INVESTIGATIONS OF SUCROSE ESTERS BY TANDEM MASS SPECTROMETRY. Terry Sumpter and Nancy J. Jensen, Philip Morris USA, Research Center, P. O. Box 26583, Richmond, Virginia 23261-6583 USA

Sucrose esters (SE) have been found in the green leaf cuticular waxes and trichome exudates from a variety of tobaccos and other plants. These diverse compounds are important as potential pest control agents and flavor precursors. This work describes the application of four-sector tandem mass spectrometry to the characterization of the SE fraction isolated from the cuticular waxes of Galpao comun tobacco. MS/MS analysis reveals complex but distinctive fragmentation patterns of the constituent SE's with fragmentation being greatly influenced by collision energy. The choice of fast atom bombardment (FAB) matrix dramatically influences the resulting mass spectrum with an interesting $[M + 8]^+$ ion observed when m-nitrobenzyl alcohol is used. This species is chemically (covalently) distinct from the corresponding SE. Cationization by Li^+ enhances the abundance of pseudomolecular ions for the SE's, and it appears that Li^+ attachment occurs exclusively at the fructose moiety. Negative-ion FAB has proven useful as well, due primarily to the reduced chemical background observed in this mode of analysis.

2028896861

18
9:40 a.m.
Wednesday

INCORPORATION OF ^{3}H-NICOTINIC ACID INTO ALKALOIDS OF BURLEY 21, LA BURLEY 21 AND *N. GLAUCA*. Franklin F. Fannin, Nicole Nicholas and Lowell P. Bush, University of Kentucky, Department of Agronomy, Lexington, Kentucky 40546-0091 USA

In vivo alkaloid biosynthesis was studied in seven day old intact seedlings or in older detached roots and leaves. The incorporation rate of ^{3}H-nicotine acid in the presence of methylputrescine into nicotine was greatest near the root tip. Very little alkaloid synthesis was detected in shoot tissues. With a four hour incubation, nicotinic acid was incorporated most rapidly into nicotine and rates of incorporation were similar for the three genotypes even though nicotine levels of the seedlings were significantly different. Only negligible amounts of nicotinic acid were incorporated into anabasine and anatabine in the *N. tabacum* genotypes; whereas, in *N. glauca* incorporation into anabasine and anatabine was 15% and 11%, respectively, of that in nicotine. These results were obtained even though nicotine and anabasine concentrations were nearly equal in the *N. glauca* seedlings. These results may mean that the availability of exogenous nicotine acid is rate-limiting and similar or more likely that the nicotine synthase activity in seedlings of the three genotypes is similar when appropriate substrates are added.

10:00 a.m. INTERMISSION

19
10:30 a.m.
Wednesday

QUANTITATIVE CHANGES OF CIGARETTE SMOKE COMPONENTS DURING A PUFF. Hideki Takahashi, Yukio Sone, Masao Matsukura and Takeshi Sakaki, Japan Tobacco Incorporated, Tobacco Science Research Laboratory, 6-2 Umegaoka, Midori-ku, Yokohama 227, Japan

This study investigated quantitative changes of smoke components during one puff. The smoke sample was 2 ml of the fourth puff taken under the following two smoking conditions: 35 ml/2 seconds and 35 ml/4 seconds. Smoke samples at five time divisions of one puff were fed directly into a multidimensional gas chromatograph (MDGC). The MDGC analyzed vapor phase components and semivolatile components of smoke simultaneously. We measured the peak areas of 51 identified compounds. The patterns of quantitative changes during the first to the fifth time division were examined for each peak with the t-test method. Under both smoking conditions, all 51 compounds produced eight patterns. For the two-second puff, the patterns were classified into three

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

groups with changes noted from the first to the third time division. The compounds contained in the three groups shared similar chemical structures and generating processes. When the cigarette was puffed for four seconds, the quantity of almost all compounds increased from the first to the second time division. Consequently, we classified the patterns into three groups with changes observed from the second to the fourth time division. With the compounds contained in each group, this classification was similar to the two-second puff. From the results described above, it was confirmed that the patterns of time-dependent quantitative changes during one puff are closely related to their generating processes and the cigarette burning temperatures.

20
10:50 a.m.
Wednesday

DETERMINATION OF CADMIUM IN CIGARETTE SMOKE AND ITS POSSIBLE ROLE AS BIOMARKER. Klaus D. Brunnemann, Jonathan E. Cox and Dietrich Hoffmann, American Health Foundation, 1 Dana Road, Valhalla, New York 10595 USA

The amount of cadmium in tobacco depends on the variety and origin of the plant, ranging from 0.2 ppm to 3.5 ppm, while its concentration in most commercial cigarette tobacco is between 1 and 2 ppm. Reported transfer rates of Cd into the mainstream (MS) and sidestream smoke range from 4% to 20% and from 50% to 58%, respectively. It was the goal of this study to determine the levels of Cd in MS and to evaluate the possible role of Cd as biomarker by measuring Cd levels in the hair of smokers and nonsmokers. For the analysis of MS, one cigarette was smoked onto a prewashed Cambridge filter. The filter pad was heated in nitric acid for two hours at 50°C, then diluted with water and analyzed by graphite furnace-atomic absorption (AA). Hair samples (25 to 72 mg), taken close to the scalp from the occipital region, were digested with nitric acid, diluted with water and analyzed by AA. A 1% monobasic ammonium phosphate solution was used as matrix modifier. Cd levels in MS ranged from 0.05 to 0.28 µg/cigarette. In a preliminary study we found 0.29 ppm (range: 0.017 to 0.99) in nonsmokers' hair (n = 15) and 0.52 ppm (range: 0.035 to 2.82) in smokers' hair (n = 15). The difference in the sample population was statistically significant ($p < 0.05$). Cd uptake is not limited to cigarette smoking and so far we have not adjusted for dietary, occupational and environmental factors. Additional data will be presented and their significance will be discussed.

2989688202

21
11:10 a.m.
Wednesday

DETERMINATION OF ALDICARB, ALDICARB SULFOXIDE AND ALDICARB SULFONE IN TOBACCO USING HPLC WITH DUAL POST-COLUMN REACTION AND FLUORESCENCE DETECTION. S. S. Yang and I. Smetena, Philip Morris USA, Research Center, P. O. Box 26583, Richmond, Virginia 23261-6583 USA

Aldicarb and its sulfoxide and sulfone metabolites were extracted from tobacco samples with organic solvents (e.g., methanol) at ambient conditions. The extract was treated with a simple procedure and then injected into an HPLC equipped with a dual post-column reaction system and a fluorescence detector. Chromatographic separation was performed on a C-18 column with a mixture of methanol/water as the mobile phase. Aldicarb residues in the eluate were hydrolyzed in the first post-column reactor with concentrated alkalis at an elevated temperature to yield methylamine and subsequently derivatized in the second post-column reactor with o-phthalaldehyde prior to fluorescence detection. The fluorescence detector was set at 330/465 nm (excitation/emission). Recoveries of aldicarb residues from tobacco samples were studied. Parameters of the HPLC and the post-column reaction system also were evaluated and will be described in detail.

22
11:30 a.m.
Wednesday

NICOTINE AND CARBOXYLIC ACIDS: STRUCTURE AND SOLUTION DYNAMICS, A MULTINUCLEAR NMR STUDY. William S. Caldwell, William M. Coleman and Patricia Martin, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

Investigations of the reaction between nicotine and carboxylic acids have been the subject of many reports. It has been proposed that nicotine forms 3:1 salts with aliphatic monocarboxylic acids such as acetic acid. These salts reportedly exist as carboxylate anions ion-paired to the dication of nicotine. We have investigated the interaction of nicotine with a variety of carboxylic acids using multinuclear NMR techniques. Levulinic acid (pK_a = 4.5) was used as a model in order to probe the interactions between nicotine and carboxylic acids. In addition, supporting data were obtained for acetic acid (pK_a = 4.75) and dichloroacetic acid (pK_a = 1.48). Titrations of nicotine with each acid were performed in aqueous and organic solutions. Proton, carbon-13, and nitrogen-15 NMR spectra of the resulting solutions revealed that diprotonation of nicotine occurred only in the presence of dichloroacetic acid. Nuclear Overhauser effect

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

spectroscopy was used to determine the degree of ion pairing between the nicotinium ion and the carboxylate ion. In water, a 3:1 ratio of nicotine and levulinic acid exists as solvent separated ions; however, in nonpolar organic solvents it exists as loosely associated ion pairs. Thorough charcterization of the species formed as a result of nicotine's interaction with these carboxylic acids will be described.

11:50 a.m. LUNCH

2028898683

WEDNESDAY MORNING

Session B

Larry W. Renfro, Presiding

23
8:40 a.m.
Wednesday

A MATHEMATICAL MODEL OF PYROLYSIS PROCESSES IN A SMOLDERING CIGARETTE.
Peishi Chen and Sung-Chul Yi, Philip Morris USA, Research Center, P. O. Box 26583, Richmond, Virginia 23261-6583 USA

A mathematical model for the pyrolysis zone in a smoldering cigarette has been proposed. In the analysis of the cigarette pyrolysis processes, a receding burning front is defined, which has a constant temperature (~450°C) and divides the cigarette into two zones, the burning zone and the pyrolysis zone. The processes considered include the pyrolysis of virgin tobacco, evaporation of water, and internal heat transport characterized by effective thermal conductivity. Thermal conduction of cigarette paper and convective and radiative heat transfer at the outer surface were also considered. The governing partial differential equations were solved using an integral method. Model predictions of smoldering speed, or linear burn rate, temperature and density profiles in the pyrolysis zone for different kinds of cigarettes were found to agree well with experimental data in the literature. The model provides a relatively fast and efficient way to simulate the pyrolysis processes and offers a practical tool for exploring important parameters for a smoldering cigarette, such as blended tobacco composition, properties of cigarette paper, and heat flux from the burning zone to the pyrolysis zone.

24
9:00 a.m.
Wednesday

NUMERICAL RESOLUTION OF 2-D COMBUSTION FRONT PROPAGATION BY ORTHOGONAL ADAPTIVE MESH TECHNIQUE.
Sung-Chul Yi, Philip Morris Research Center, P. O. Box 26583, Richmond, Virginia 23261-6583 USA

The simulation of combustion phenomena poses a real challenge to numerical methods due to the "stiffness" of the governing equations, i.e. there is a wide variation in reaction rates present in this system. To cope with the possibility of extremely abrupt changes in the values of the solution over very short distances, adaptive mesh techniques can be applied to resolve the high activity regions by ordering grid points in appropriate places. To avoid a control volume formulation of the governing equations, a simple orthogonal, adaptive mesh technique is employed.

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

This involves separate adaptation in the x and y directions with the Lagrange polynomial and a normal cubic spline differentiator. The numerical technique is tested on the two-dimensional smoldering-cigarette model which represents a system of coupled, highly nonlinear, parabolic-hyperbolic, partial differential equations exhibiting solutions with steep gradients which move rapidly over space. Use of a simple, orthogonal, adaptive-mesh adaptive mesh technique proved to be both efficient and accurate for the simulation of a smoldering combustion cigarette.

25
9:20 a.m.
Wednesday

A COMPARATIVE IGNITION PROPENSITY STUDY VIA INFRARED IMAGING ANALYSIS. Marian Joyce, Tim Nestor and Stephen Sears, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

The literature contains several reports of infrared imaging analysis in the investigation of cigarette surface temperature. No studies have reported its use as a tool to study ignition propensity. Infrared imaging analysis offers a nonintrusive means of monitoring the surface temperature of cigarette fire cones. We have applied the technique to a set of cigarettes of varying construction parameters. The cigarettes studied revealed a broad range of ignition propensity results as determined by a mockup test adapted from the National Bureau of Standards (now National Institute of Standards and Technology, or NIST). An AGEMA Thermal Imaging System (TIS) equipped with a burst recording unit (BRU) was modified and used to study the surface temperature distributions in the cigarettes. Modifications included the addition of Ethernet Local Area Network (LAN), a remote triggering device to simultaneously activate the BRU, and a smoking machine. LAN allowed transfer of raw data files to a SUN workstation for processing, analysis and storage. Analyses of the thermal images show good agreement with assorted mockup techniques.

26
9:40 a.m.
Wednesday

ORGANIC POTASSIUM. ITS ROLE IN CONTROLLING THE RATE OF BURN AND 'TAR' CONTENT OF TOBACCO PRODUCTS. James W. Horler, Professional Services, The White House, Wookey, Wells, Somerset BA5 1NY, England; Nicolas Baskévitch, LTP Industries, Le Mans, France

One of the most important requirements in the design of tobacco products is the ability to control the burn rate of the tobacco blend. It has long been recognized that both potassium and chloride play an important part in determining

2028896864

the burn rate of tobacco but the relationships have always remained unclear. It is shown in this paper that it is not the total potassium content that is important in controlling burn rate but the quantity of potassium present as organic acid salts. In other words changing the potassium chloride content of the tobacco will have no effect upon burn rate but changing the potasssium malate content, for example, will have a considerable effect. The quantity of organic potassium present in a tobacco product can be calculated by subtracting the potassium associated with the chloride and sulphate in the product from the total potassium present. The relationship between organic potassium and burn rate has been established for a wide range of reconstituted and natural tobaccos and even, for test purposes, pure cellulose. It has been found for all these products that the burn rate increases as the organic potassium content increases to a peak at about 2.5% K_2O and then the burn rate reduces. Tobacco products with negative values of organic potassium either will not burn or burn very poorly. As the organic potassium increases in a tobacco product there is a simple linear reduction in the 'tar' content and in the other smoke constituents. There is no evidence of a peak at 2.5% K_2O as is found with rate of burn. The burn rate and level of smoke constituents of reconstituted and natural tobacco products can be readily modified by the addition or reduction in the quantity of organic potassium salts present in the blend.

10:00 a.m. INTERMISSION

27
10:30 a.m.
Wednesday

THE EFFECT OF CELLULOSE ACETATE FIBER AND FILTER PARAMETERS ON ROD FIRMNESS. Cheryl E. Johnson, Hoechst Celanese Corporation, P. O. Box 32414, Charlotte, North Carolina 28232 USA

Cigarette filter firmness has become a greater concern as the cigarette industry has shifted toward lower denier per filament and total denier tows. An earlier study determined that these trends would have a deleterious effect on filter firmness, as would decreasing the effective crimp index, decreasing the plasticizer level, or increasing the filter circumference. However, this study included a limited number of tow items between 3.5 and 6.5 dpf and 35,000 to 45,000 total denier. The updated study examined a broader range of tow items (1.8 to 8.0 dpf and 25,000 to 50,000 total denier) which more accurately represented the tow item mix used today. In addition, the circumference ranges tested were tied to the total denier of each tow item, resulting in a realistic range of circumferences and firmness levels. The new predictive model is presented along with several practical examples of the impact of filter and tow parameters on firmness.

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

28
10:50 a.m.
Wednesday

THE ROLE OF PAPER FILTERS IN THE CHANGING CIGARETTE MARKET. Richard J. K. Shepherd, FIL International Limited, 4-6 Arden Grove, Harpenden, Hertfordshire, England

The growing awareness around the world of the need to be more sensitive to the environment we live in is giving rise to pressure within the tobacco industry to ensure that it too plays its part in this debate. In addition to ensuring that the manufacturing processes are as environmentally friendly as possible, it is increasingly relevant to consider the effect of discarded cigarette ends on the environment. Clearly, the cigarette filter material plays an extremely important part in this and a renewed role for paper in this respect will be proposed. The performance characteristics of paper in terms of its 'tar' and nicotine retention are described and the implications of this for cigarette design are considered. Data will be presented which compare the effect of paper and cellulose acetate filters on the semivolatile fraction of tobacco smoke as these are considered to affect the taste of cigarettes. Attempts to modify the effect that paper filters have on the semivolatiles so that it matches the effect of cellulose acetate will also be described. Finally, data will be presented which show that under the conditions of the modified Sturm Test for ready biodegradability, paper filters degrade significantly faster than cellulose acetate.

29
11:10 a.m.
Wednesday

PREDICTION OF FILTER CIGARETTE DESIGN UNDER RESTRICTED CONDITIONS. Young-Hoh Kim, Kwang-Soo Rhim and Yong-Tae Kim, Korea Ginseng and Tobacco Research Institute, #302, Shinsungdong, Yoosungku, Daejeonshi, Korea 305-345

To minimize the time ordinarily spent in mono filter cigarette design, we studied the relationship between seven major independent variables; filament (X_1), total denier (X_2), porosity of the filter plug wrap (X_3), filter length (X_4), porosity of the tip paper (X_5) and cigarette paper (X_6), and filling weight of the reference cut tobacco (X_7). Ninety trial numbers were obtained as a result of using rotatable central composite design and the results were analyzed by stepwise multiple regression analysis using SAS/pc. Those equations obtained were as follows:

$$UPD(Y_1) = 82.96 - 3.80X_1 + 2.50X_2 - 3.29X_3 - 3.15X_5 - 0.83X_2^2 + 1.88X_5X_6 - 1.38X_5X_7 \quad (R^2 = 0.63)$$

2028896865

$$EPD(Y2 = 120.91 - 5.70X_1 + 3.60X_2 + 4.23X_4 - 0.93X_6 + 4.06X_7 \quad (R^2 = 0.84)$$

$$TVR(Y_3) = 49.70 - 0.78X_1 + 3.60X_3 + 2.00X_4 + 4.20X_5 - 0.93X_6 + 2.64X_7 - 1.07X_1X_2 + 1.01X_1X_3 + 1.05X_2X_6 + 0.45X_3^2 - 0.64X_4^2 + 1.29X_4X_6 - 0.97X_4X_7 - 1.28X_5X_6 + 1.53X_5X_7 + 1.39X_6X_7 \quad (R^2 = 0.65)$$

$$EVR(Y_4) = 3.24 - 0.21X_3 - 0.20X_4 - 0.24X_5 + 0.67X_6 + 0.26X_4X_7 \quad (R^2 = 0.55)$$

Where, UPD (unencapsulated pressure drop), EPD (encapsulated pressure drop), TVR (tip ventilation rate), EVR (envelope ventilation rate) and other equations for smoke components ('tar', nicotine, carbon monoxide) were also established.

30
11:30 a.m.
Wednesday

THE EFFECT OF VENTILATION ON BURN RATES. Lydia J. Holt and Larry W. Renfro, Eastman Chemical Company, P. O. Box 1972, Kingsport, Tennessee 37662-5150 USA

A complete understanding of how changes in ventilation affect cigarette performance is necessary to determine the optimum filtration system for ventilated cigarettes. One important aspect of ventilation is the effect on the tobacco column burn rate which affects the puff count and the availables generated during smoking. Computer image analysis was used to measure the position of the burn line on the tobacco column before and after each puff for each cigarette on a 20-port rotary smoking machine. Burn rates during puffs (dynamic) and between puffs (static) were then calculated individually for each cigarette. Burn rates were measured on commercial cigarettes which had been altered to provide a wide range of ventilation levels without changing the tobacco column or filter components. The results show the effect of static ventilation on burn rates and the changes in burn rate with changing ventilation as the tobacco column is consumed. By relating the static and dynamic burn rates to the availables, a model can be produced to predict the tobacco column deliveries for each puff of the cigarette.

11:50 a.m. LUNCH

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

WEDNESDAY AFTERNOON

Joint Session

R. Hugo Martin, Presiding

31
1:40 p.m.
Wednesday

SOLUTION CHEMISTRY OF PSEUDOOXYNICOTINE AND 2'-CYANONICOTINE. A HIGH FIELD NMR STUDY. Grayland P. Dobson and William S. Caldwell, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

Pseudooxynicotine (PON), a major metabolite of nicotine in certain soil bacteria and fungi, exists in equilibrium with nicotine-1',2'-iminium ion in aqueous solution. A recent report that nicotine-1',2'-iminium ion (detected as the cyanide adduct) was present in smokers' urine led to speculation that PON may also be a human metabolite of nicotine. Using proton and carbon NMR, we have confirmed that the equilibrium between PON and nicotine-1',2'-iminium ion in aqueous solution is pH dependent. We also used NMR to investigate some additional chemical properties of PON and 2'-cyanonicotine. When a basic solution of PON was extracted with methylene chloride, the organic phase was found to contain N-methylmyosmine. Upon removal of the organic solvent and addition of water, an equilibrium mixture of PON and nicotine-1',2'-iminium ion was obtained and N-methylmyosmine was no longer detected. When an organic solution of N-methylmyosmine was allowed to stand at room temperature, it rapidly decomposed to unidentified products. When a basic solution of PON was treated with a slight excess of KCN, 2'-cyanonicotine was rapidly and quantitatively formed. It was found to be stable between pH 9 and 10. 2'-Cyanonicotine could be quantitatively extracted into methylene chloride resulting in a stable organic solution. These observations form the basis of a method for the quantitative determination of PON in aqueous solutions.

32
2:00 p.m.
Wednesday

DETERMINATION OF NICOTINE-1',2'- AND 1',5'-IMINIUM IONS IN AQUEOUS SOLUTIONS. Gary D. Byrd, G. M. Dull, Grayland P. Dobson and William S. Caldwell, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

The major pathway for nicotine metabolism in humans involves cytochrome P-450 catalyzed oxidation of the 5' position of

2028896899

the pyrrolidine ring to form nicotine-1',5'-iminium ion. Pseudooxynicotine (PON), a major metabolite of nicotine in certain soil bacteria and fungi, exists in equilibrium with nicotine-1',2'-iminium ion in aqueous solution. A recent report that both nicotine-1',5'- and nicotine-1',2'-iminium ions were present in smokers' urine led to speculation that PON may also be a human metabolite of nicotine. Direct quantitation of these iminium ions in aqueous solutions is complicated by their extreme polarity and instability to traditional extraction methods. We have developed a method for quantifying nicotine-1',5'- and nicotine-1'-2'-iminium ions in aqueous solutions which involves derivatization with KCN to form 2'- and 5'-cyanonicotines. Aqueous samples were spiked with the internal standard methyl-d_3-PON, adjusted to pH 10, treated with excess KCN, and extracted with CH_2Cl_2. The organic fraction was reduced in volume and analyzed by GC/MS using a DB5 capillary column. LOD's for the two analytes were approximately 10 ng/ml. The utility of this method for quantifying nicotine-1',5'- and -1',2'-iminium ions in smokers' urine will be discussed.

33
2:20 p.m.
Wednesday

IDENTIFICATION AND QUANTIFICATION OF TOBACCO-SPECIFIC NITROSAMINE PRECURSORS IN AIR-CURED TOBACCO. Harold R. Burton and Xiaochen Wei, University of Kentucky, Department of Agronomy, Lexington, Kentucky 40546-0091 USA; William S. Caldwell, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

In recent years there has been interest in identifying the factors that contribute to the accumulation of tobacco-specific nitrosamines (TSNA) during curing and processing of tobacco. Of particular interest is the accumulation of 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK) during curing. From recent kinetic data, it has been assumed that the level of pseudooxynicotine [4-(methylamino)-1-(3-pyridyl-1-butanone)], in part, limits the accumulation of NNK during the curing process. Direct quantification of pseudooxynicotine (PON) has been elusive since it undergoes intramolecular cyclization to yield 2'-hydroxynicotine which undergoes dehydration yielding 2',3'-dehydronicotine (methylmyosmine) during conventional extraction techniques. A method has been developed to indirectly quantify PON as 2'-cyanonicotine by treatment of the alkaline extract of tobacco with excess KCN. 2'-Cyanonicotine in the methylene chloride extract was quantified by GC/MS using a 15 M by 0.25 mm ID capillary column coated with 0.25μ DB5 liquid phase. The internal standard of choice was trideuterated PON since it allowed for the quantification of 5-cyanonicotine using selected ion monitoring. This method not only allows for the quantification of PON but for the

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

analyses of 3'-pyridyl-3-methylaminobutyraldehyde. Analytical data from a curing study will be presented to show the concentration ranges of PON that occur during the air-curing process.

34
2:40 p.m.
Wednesday

DIFFERENCES IN COMPOSITION OF AIR-CURED BURLEY TOBACCO USING MODIFIED FIELD CURING STRUCTURES. 1. CHANGES IN NITROGENOUS CONSTITUENTS. Harold R. Burton and N. K. Dye, University of Kentucky, Department of Agronomy, Lexington, Kentucky 40546-0091 USA; L. Walton, University of Kentucky, Department of Agricultural Engineering, Lexington, Kentucky 40546-0091 USA

In 1992, a study was initiated to determine if the composition of burley tobacco was different when it was air-cured in temporary field structures versus conventional barn cured burley tobacco. The study consisted of four different curing regimes for the field cured structures, conventional barn curing and environmental chamber curing (24°C and 70% RH). Samples from this study for the six curing environments were replicated four times. The 1992 growing and curing seasons in the Lexington area were atypical of the norm and resulted in increased microbial activity that resulted in lower quality air-cured burley tobacco. This atypical curing season provided samples that allowed for determining the influence of adverse curing conditions on the composition of air-cured burley tobacco. Recent studies in our laboratory have shown poor air-curing environments resulted in increased levels of nitrite and tobacco-specific nitrosamines (TSNA). Samples from these studies were analyzed for nitrite-N, nitrate-N, individual alkaloids, total nitrogen, and TSNA. Nitrite-N values ranged from 2 to 4,400 $\mu g\ g^{-1}$ while TSNA values ranged from 3 to 125 $\mu g\ g^{-1}$. Statistical analyses of the data showed there was a treatment effect, stalk position effect, and plant part effect on the accumulations of individual TSNA's and nitrite-N. Data will be presented to show that the top of the plant contains the highest levels of individual TSNA's and nitrite-N and there was a positive correlation between nitrite accumulation and TSNA's.

3:00 p.m. INTERMISSION

2989688202

35
3:20 p.m.
Wednesday

NOVEL DITERPENE GLYCOSIDES FROM *NICOTIANA* SPECIES. Tetsuya Tobita and Y. Shinozaki, Japan Tobacco Incorporated, Tobacco Technology Planning Department, 1-17-7 Yokokawa, Sumida-ku, Tokyo 130, Japan; H. Takahashi, M. Mizutani and T. Matsuzaki, Japan Tobacco Incorporated, Tobacco Science Research Laboratory, 6-2 Umegaoka, Midori-ku, Yokohama, Kanagawa 227, Japan

The leaves of *Nicotiana* species contain several varieties of terpene glycosides, such as 5,6-epoxy-5,6-dihydro-3-hydroxy-β-ionyl-β-glucoside, blumenol A-β-glucoside and loliolide-β-glucoside. These compounds are considered the precursors of tobacco smoke aroma. We identified three novel diterpene glycosides from *Nicotiana tabacum* and *Nicotiana umbratica*. These compounds consisted of hydroxygeranyllinolool as an aglycone and two or three molecules of either glucose or rhamnose. The locations of aglycone and glycosides in the molecules were determined by 2D-NMR using the HMBC technique. The molecular weight was determined by FAB-MS. The structures were: (6E,10E,14Z)-20-hydroxygeranyllinalyl-3-O-α-L-rhamnosyl(1→4)-β-D-glucoside-20-O-[β-D-glucosyl(1→2)]-[α-L-rhamnoxyl(1→6)]-β-D-glucoside (I); (6E,10E,14Z)-20-hydroxygeranyllinalyl-3-O-α-L-rhamnosyl(1→4)-β-D-glucoside-20-O-[α-L-rhamnosyl(1→4)]-[α-L-rhamnoxyl(1→6)]-β-D-glucoside (II); (6E,10E,14Z)-20-hydroxygeranyllinalyl-3-O-β-D-glucoside-20-O-[α-L rhamnosyl(1→4)]-[α-L-rhamnosyl(1→6)]-β-D-glucoside-20-O-[α-L-rhamnoxyl(1→4)]-[α-L-rhamnosyl(1→6)]-β-D-glucoside (III). The distribution of these diterpene glycosides in *Nicotiana* species will be discussed.

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

36
3:40 p.m.
Wednesday

VOLATILE CONSTITUENTS FROM FLOWERS OF SELECTED *NICOTIANA* SPECIES. William S. Schlotzhauer, Robert J. Horvat and Orestes T. Chortyk, United States Department of Agricultural, Agricultural Research Service, Phytochemical Research Unit, P. O. Box 5677, Athens, Georgia 30613 USA; D. Michael Jackson, United States Department of Agricultural, Agricultural Research Service, Crops Research Laboratory, P. O. Box 1168, Oxford, North Carolina 27565-1168 USA

Volatile compounds produced during the growth cycle of tobacco and related *Nicotiana* species are believed to play a critical role in plant/insect interactions. Field observations have shown that *Cardiochiles Nigriceps*, a beneficial predator of the tobacco budworm, is preferentially attracted to certain *Nicotiana* species plants. Since flowers are an important source of volatiles, a study was performed to identify the volatile flower constituents of selected species, with differing levels of attraction, including *N. Noctiflora*, *N. Sanderae*, *N. Forgetiana*, *N. Longiflora*, *N. Alata*, and *N. Rustica*. Flowers were removed in the field and surface chemicals were extracted by dipping into methylene chloride. Extracts were stored at 0°C. Volatiles were concentrated by continuous extraction with hexane, using a modified Likens-Nickerson apparatus, and were identified by capillary gas chromatography/mass spectrometry. The different species were found to produce unique patterns of volatiles. Constituents identified include alcohols, ketones, esters, mono-terpenes, sesquiterpenes, sesquiterpene alcohols, and a number of aromatic (benzenoid) components. The similarities and differences in composition will be discussed.

37
4:00 p.m.
Wednesday

EVALUATION OF LOW NICOTINE FLUE-CURED TOBACCO IN BLENDED CIGARETTES. Willard W. Weeks, North Carolina State University, Department of Crop Science, P. O. Box 7620, Raleigh, North Carolina 27695 USA; Verne A. Sisson, United States Department of Agriculture, Agricultural Research Service, Crops Research Laboratory, P. O. Box 1168, Oxford, North Carolina 27565-1168 USA

Blended cigarettes were fabricated from burley, oriental, and low nicotine flue-cured tobaccos. The nicotine concentration of burley, oriental and low nicotine flue-cured tobacco was 3.66%, 1.16% and 0.92% to 1.65%, respectively. Each blend was made up of 8 grams of

2028896868

oriental, 15 grams of burley and flue-cured was added until the nicotine concentration of the blend was diluted to 2%. To obtain 2% nicotine, as little as 18 grams and as much as 55 grams of flue-cured tobacco was needed to complete individual blends. Ten filtered cigarettes, from each blend, made with a handmaking cigarette machine were evaluated by a three man smoke panel. Each cigarette was evaluated for balance, impact, off-flavor and irritation. Panelists ranked the cigarettes made from blends in order of their personal preference. Cigarettes from blends made from flue-cured tobacco with 1.55%, 1.38%, and 1.63% nicotine were preferred by all panelists, and cigarettes made from flue-cured tobacco with 1.65%, 1.10%, and 0.94% nicotine levels were ranked last. Due to the variation in nicotine concentration among the flue-cured tobaccos used in the blends, reducing sugar concentration of blends ranged from 10% to 24%. Volatile oils were measured from individual tobaccos and tobacco blends. The effect of chemistry from individual tobaccos and tobacco blends upon smoke perception will be discussed.

38
4:20 p.m.
Wednesday

NATURAL PRODUCTS AS SOURCES OF AROMA AND FLAVOR INGREDIENTS. William M. Coleman, J. L. White, T. A. Perfetti and W. T. Morgan, R. J. Reynolds Tobacco Company, Bowman Gray Technical Center, P. O. Box 1487, Winston-Salem, North Carolina 27102 USA

A better chemical understanding of the Maillard Reaction has been developed which describes a molecular basis for the production of flavorful, volatile aromatic compounds from the reactions of and between amino acids and sugars at an elevated temperature ("roasting"). By employing a variety of natural products, a clearer understanding of the common mechanisms for the production of many important flavorants and odorants has been established. Regardless of the starting natural product, the same qualitative profile of volatile materials is produced upon "roasting" an aqueous extract of the natural products. The notable differences in taste and aroma arise not from significant differences in the qualitative nature of the materials but from variations in the quantitative distribution of the volatile materials produced. By making use of: (a) the similarity in the qualitative nature of the "roasted" materials, (b) dissimilarities in quantitative distribution of the volatile materials and (c) computer linear combination equation capabilities, it has been possible to produce an artificial peanut flavor from a combination of non-peanut containing natural products.

4:40 p.m. ADJOURNMENT

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

4:50 p.m. BUSINESS MEETING

5:20 p.m. TOBACCO SCIENCE COUNCIL MEETING

2028898698

THURSDAY MORNING

Joint Session

Bryon F. Price, Presiding

9:00 a.m. Philip Morris Awardee, Research Highlights

39
9:50 a.m.
Thursday

A NEW ANALYTICAL METHOD FOR THE DETERMINATION OF NITRITE IN NANOGRAM LEVELS IN TOBACCO. Alex Christakopoulos, Ann Palmqvist and Inger Wahlberg, Reserca AB, S-118 84 Stockholm, Sweden

Nitrite occurs naturally both in plants and animals. While present in trace amounts only in green tobacco, concentrations up to 1,000 ppm or more may be found in cured and aged tobacco. Since nitrite may react with secondary or tertiary amines to form N-nitroso compounds with potential carcinogenic, mutagenic or toxic properties, it is of importance to measure its concentration in tobacco in an accurate manner. We now report the development of a new sensitive and selective method for qualitative and quantitative analysis of nitrite in tobacco samples. The inorganic nitrite present in the tobacco sample is reacted with ethanol under acidic conditions, capped autosampler vials being used as the reaction vessel. The ethyl nitrite formed, which is present in the headspace, is quantified by combined headspace/gas chromatography-mass spectrometry using the selected ion monitoring technique. ^{15}N-Sodium nitrite is added to the tobacco sample as the internal standard in order to compensate for errors caused by instrumentation and incomplete recovery during the derivatization procedure. The detection limit is 0.05 µg/gram of tobacco. The method, which has been automated, has been applied successfully for the determination of nitrite in tobacco, vegetables and water.

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

40
10:10 a.m.
Thursday

INFLUENCE OF TOBACCO NITRATE CONTENT ON THE YIELDS OF SELECTED MAINSTREAM SMOKE COMPONENTS. Anthony R. Tricker, Verband der Cigaretten Industrie, Königswinterer Str. 550, 5300 Bonn 3, Germany; Gerhard Scherer and F. Adlkofer, Analytisch-Biologisches Forschungslabor, Goethestr. 20, 8000 München 2, Germany

Machine-made nonfilter experimental cigarettes with a tobacco filler nitrate content of 0.22%, 0.53%, 1.12% and 1.78% were produced using a blend of 50% Virginia, 15% oriental, 10% burley and 25% reconstituted sheet tobacco containing added potassium nitrate. The mainstream smoke deliveries were determined for condensate, nicotine, carbon monoxide (CO), nitrogen oxides (NO_x), nitric oxide (NO), hydrogen cyanide (HCN), N-nitrosodimethylamine (NDMA), N-nitrosoethylmethylamine (NEMA), N-nitrosopyrrolidine (NPYR), N-nitrosonornicotine (NNN), N-nitrosoanatabine (NAT), 4-(N-nitrosomethylamino)-4-(3-pyridyl)-1-butanone (NNK), 2-phenylnaphthalene, fluoranthene, benzo[a]anthracene, pyrene, chrysene, benzo[a]pyrene, benzo[e]pyrene, perylene, dibenz-[a,h]anthracene, harman, norharman, formaldehyde, acetaldehyde, acrolein, propionaldehyde, phenol, catechol, cresols, 2-methoxyphenol and 3,4-dimethoxyphenol. Positive correlations between increasing the nitrate concentration in the tobacco filler and mainstream smoke yields of NO_x ($r = 0.998$), NO ($r = 0.997$), NEMA ($r = 0.993$), NPYR ($r = 0.958$), NAT ($r = 0.914$), total volatile and tobacco-specific N-nitrosamines ($r = 0.886$) were found. Negative correlations were found for catechol ($r = 0.921$) harman ($r = 0.918$) and norharman ($r = 0.918$). Total polycyclic aromatic hydrocarbons showed a negative correlation ($r = 0.972$) while positive correlations were found for benzo[a]pyrene ($r = 0.947$), benzo[e]pyrene ($r = 0.839$) and perylene ($r = 0.957$). Data from this study will be presented and compared to that obtained for the 2R1 Kentucky reference cigarette.

10:30 a.m. INTERMISSION

0289688202

41
10:50 a.m.
Thursday

A METHOD FOR THE DETERMINATION OF POLYPHENOLS IN TOBACCO BY HPLC AND ITS APPLICATION TO THE ANALYSIS OF VARIOUS TOBACCO TYPES. Steven J. Stotesbury, British-American Tobacco Company Limited, BAT Company Technology Centre, Regents Park Road, Southampton SO9 1PE, England

Polyphenols are a loosely related group of compounds which in tobacco comprise the compounds rutin, scopoletin, and chlorogenic acid and its isomers. They are destroyed by air-curing and, therefore, occur in very low concentrations in burley and Maryland tobaccos. In oriental and Turkish tobaccos, the overall polyphenol concentration is intermediate to that of the air- and flue-cured varieties but with a different relative proportion of chlorogenic acid and rutin to Virginia tobaccos. While looking at an analytical method based on high performance liquid chromatography (HPLC), it was discovered that caffeic acid elutes near to one of the caffeoylquinic acid isomers. This would appear to have caused ambiguities in the interpretation of chromatograms reported by previous workers. The peak separation has been greatly improved by employing a nonlinear gradient elution program as part of the method. Chromatograms from typical examples each of a burley, Virginia and oriental blend using our improved method will be presented. The method has also been applied to the analysis of smoke condensates to see whether any of the polyphenols transfer to the smoke. Smoke condensate was collected on Cambridge filter pads, extracted with methanol and analyzed by HPLC. The results indicate that up to 15% of scopoletin transfers from tobacco to the smoke.

42
11:10 a.m.
Thursday

THE POLYPHENOLS OF *NICOTIANA*. Maurice E. Snook, Ray F. Severson and Orestes T. Chortyk, United States Department of Agricultural, Agricultural Research Service, Richard B. Russell Agricultural Research Center, P. O. Box 5677, Athens, Georgia 30613 USA; Verne A. Sisson, United States Department of Agricultural, Agricultural Research Service, Crops Research Laboratory, P. O. Box 1168, Oxford, North Carolina 27565-1168 USA

Polyphenols are important tobacco constituents that affect the final properties and qualities of the cured leaf. We have determined the polyphenols of approximately 1,500 tobacco introductions (TI), classified as flue-cured, burley, oriental, cigar, primitive, pale yellow, and TI448

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

type, which were grown at four locations in North Carolina (Oxford and Kinston - primitive, Whiteville - oriental, and Reidsville - cigar) in 1988. Fresh tissue from each variety (stored in methanol) was analyzed by HPLC for polyphenols (caffeoylquinic acids, CQA; rutin; and kaempferol-3-rutinoside, K-3-Ru). Flue-cured, burley, and oriental TI's were very similar in total CQA (averaging 0.35% fresh weight) and total polyphenols (0.43%), while cigar types had half of these levels and pale yellow, primitive, and TI448 types had slightly higher levels (CQA - 0.43%, total - 0.54%). Rutin levels were relatively stable in all types (0.04% to 0.08%) except primitive (0.15%). Polyphenols were also determined in freeze-dried mature leaves of 62 *Nicotiana* species grown in the greenhouse (2 years) and the field (3 years), and in the flowers, bud leaves, and roots of the field grown species. In mature species leaves, total CQA content remained relatively constant in greenhouse versus field, while rutin levels increased from twofold to tenfold. Only rutin and K-3-Ru were found in the TI's, while the species contained many other flavonol glycosides; flowers, in particular, being the most varied. Flowers, bud leaves and the roots were particularly rich in phenolic amides. These determinations should aid in breeding studies for varying the amounts of polyphenols.

43
11:30 a.m.
Thursday

AN IMPROVED INSTRUMENT AND DATA ANALYSIS APPROACH FOR THE STUDY OF HOT COLLAPSE OF CIGARETTE FILTERS. Lance J. Deutsch, Hoechst Celanese Corporation, P. O. Box 32414, Charlotte, NC 28232 USA

The subject of hot collapse (the term given the propensity of a cigarette filter to irreversibly deform during smoking) has been studied by researchers for several years. A proposed method for quantifying hot collapse was published by a CORESTA Task force in 1991. The task force made a number of recommendations for improving the test instrument before it disbanded. The ability to study and quantify hot collapse remains a need in the Hoechst Celanese Corporation filter product laboratories. Using the recommendations of the CORESTA Hot Collapse Task Force as a starting point, a new instrument was developed from the remnants of the old. The new instrument has state-of-the-art sensing devices to determine filter depression and length of tobacco column burned. Instrument control and data acquisition are achieved via an IBM PC computer with an easy-to-use interface. In addition, an increased data acquisition rate coupled with the ability to continuously determine the amount of tobacco burned provides a much more detailed account of the hot collapse phenomenon. This, in turn, allows sophisticated curve fitting and other statistical tools to be applied toward building a model for hot collapse. The instrument and the data analysis techniques will be discussed.

2028896871

11:50 a.m. ADJOURNMENT

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

AUTHOR INDEX
(by abstract number)

Adlkofer, F.	8, 40
Andersson, G.	13
Axéll, T.	13
Baskévitch, N.	26
Best, F. W.	6
Borgerding, M. F.	4
Borschke, A. J.	2
Brunnemann, K. D.	12, 20
Burton, H. R.	33, 34
Bush, L. P.	18
Byrd, G. D.	32
Caldwell, W. S.	22, 31, 32, 33
Chen, P.	23
Chortyk, O. T.	36, 42
Christakopoulos, A.	39
Coggins, C. R. E.	11
Coleman, W. M.	22, 38
Cox, J. E.	20
Curvall, M.	13
Davis, R. A.	7, 14
Deutsch, L. J.	43
Djordjevic, M. V.	12
Dobson, G. P.	31, 32
Dong, J. Z.	16
Dull, G. M.	32
Dunlap, S. P.	15
Dye, N. K.	34
Fan, J.	12
Fannin, F. F.	18
Heavner, D. L.	7
Hege, R. B.	7
Hicks, R. D.	15
Hoffmann, D.	12, 20
Holt, L. J.	30
Hopp, R.	1
Horler, J. W.	26
Horvat, R. J.	36
Jackson, D. M.	36
Jensen, N. J.	17
Johnson, C. E.	27
Joyce, M.	25
Kaiserman, M. J.	9, 10
Kim, Y. H.	29
Kim, Y. T.	29
Kossien, I.	8
Lauterbach, J. R.	16
Maiolo, K. C.	7
Martin, P.	22
Matsukura, M.	19
Matsuzaki, T.	35
Mizutani, N.	35
Moldoveanu, S. C.	16
Morgan, W. T.	7, 38

2028696872

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

AUTHOR INDEX (continued)
(by abstract number)

Nestor, T.	25
Nicholas, N.	18
Ogden, M. W.	7
Palmqvist, A.	39
Perfetti, T. A.	38
Reid, J. R.	3
Renfro, L. W.	30
Rhim, K. S.	29
Rickert, W. S.	9, 10
Riedel, K.	8
Ruppert, T.	8
Ruzeskovic, D.	13
Sakaki, T.	19
Scherer, G.	8, 40
Schlotzhauer, W. S.	36
Sears, S.	25
Severson, R. F.	42
Shepherd, R. J. K.	28
Shinozaki, Y.	35
Sisson, V. A.	37, 42
Smetena, I.	21
Snook, M. E.	42
Sone, Y.	19
Stiles, M. F.	7, 14
Stotesbury, S. J.	41
Sumpter, T.	17
Takahashi, H.	19, 35
Tobita, T.	35
Tricer, A. R.	40
Wahlberg, I.	39
Walton, L.	34
Weeks, W. M.	37
Wei, X.	33
White, J. L.	38
Wilson, S. A.	5
Yang, S. S.	21
Yi, S. C.	23, 24

2028896873

NOTES

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

NOTES

2028896874

Tobacco Chemists' Research Conference - Volume 47

NOTES

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

NOTES

2028896875

47th TOBACCO CHEMISTS' RESEARCH CONFERENCE
October 18-21, 1993
Gatlinburg, Tennessee

Donald J. Fowlkes, Chairperson

PROGRAM EDITORIAL COMMITTEE

S. Wayne McCarty, Chairperson
Michael W. Ogden
Harold R. Burton

POLICY COMMITTEE

Maurice Snook, Chairperson

Bill Owens	Charlie Green
Dallas Pinion	Lowell Bush

LOCAL ARRANGEMENTS COMMITTEE

P. P. Hunter, Chairperson

E. E. Burgess	J. T. Matthews
J. E. Foss	R. D. Miller
C. H. Hadden	S. D. Mundy
W. A. Krueger	C. D. Pless

GUEST PROGRAM COMMITTEE

Stella Hunter, Chairperson

Millie Burgess	Betty Krueger
Jan Foss	Linda Matthews
Carol Fowlkes	Karen Miller
Marge Gossett	Sandy Mundy
Katherine Hadden	Doris Pless
Phyllis Klindt	Becky Sewell

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000

48th TOBACCO CHEMISTS' RESEARCH CONFERENCE
Sepember 25-28, 1994

Holiday Inn Four Seasons
Greensboro, North Carolina

Dr. Larry H. Gains, Chairperson
Lorillard Tobacco Company
420 English Street
Greensboro, NC 27420 USA
Telephone: (919) 373-6745
Fax: (919) 373-6640

2028896876

Source: https://www.industrydocuments.ucsf.edu/docs/ztcl0000